IS : 5762 - 1970
(Reaffirmed 1998)
(Reaffirmed 2015)

Indian Standard

METHODS FOR DETERMINATION OF MELTING POINT AND MELTING RANGE

(First Reprint SEPTEMBER 1978)

UDC 536.421.1

INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

March 1971

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INDIAN STANDARDS INSTITUTION
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METHODS FOR DETERMINATION OF MELTING POINT AND MELTING RANGE

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 8 August 1970, after the draft finalized by the Chemical Standards Sectional Committee had been approved by the Chemical Division Council.

0.2 The melting point is a characteristic physical constant of the individual material. According to the melting point it is possible to evaluate the purity of the tested material. Moisture in water soluble materials lowers the melting point. It is, therefore, necessary to dry the material thoroughly before the determination of melting point. It is likewise necessary to drive off the solvent from the material which contains the crystallizing liquid.

0.3 In the preparation of this standard assistance derived from doc No. 69/3898 'Draft British Standard method for determination of melting point and/or melting range' prepared by British Standards Institution, London; is acknowledged.

0.4 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it should be done in accordance with IS : 2-1960*.

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1. SCOPE

1.1 This standard prescribes methods for determination of melting point and melting range of materials.

1.1.1 The standard covers the determination of melting point and melting range for materials which exist as solids or liquids at ordinary temperatures.

2. TERMINOLOGY

2.1 Melting Point — It is the temperature at which liquefication of the material occurs, as indicated by the formation of a definite meniscus.

*Rules for rounding off numerical values (*revised*).

2.2 Melting Range — The range between the temperature at which the substance collapses or forms droplets on the wall of the capillary tube, and the temperature at which it is completely melted as shown by the disappearance of the solid phase.

3. PRINCIPLE

3.1 The solid material is heated in a capillary tube in a device under prescribed conditions.

3.2 The liquid material is frozen and allowed to warm up.

4. APPARATUS

4.1 Heating Bath — of suitable glass, of suitable construction and capacity and of such height as to contain not less than 140 mm depth of liquid. The bath should be provided with a suitable stirrer capable of rapidly mixing the liquid and heated by a controllable source of heat. The use of a draught screen is recommended. In place of heating bath, Thiel's tube may also be used.

NOTE — Silicone oil is normally recommended as it shows excellent temperature resisting properties. Liquid paraffin may be used for temperatures below 250°C. Glycerol or glycol is suitable for temperatures below 200°C. A solution of potassium sulphate in concentrated sulphuric acid is suitable for higher temperatures.

4.2 Electrically Operated Melting Point Apparatus — any suitable and commercially available one.

4.3 Capillary Tubes — Clean a length of the glass tubing, with a mixture of chromic and sulphuric acids, wash with water and dry. Draw the dried tubing into capillary of about 1 mm bore and as uniform as possible. Divide the tubing into 7 to 10 cm lengths by sealing it off in the flame. Keep the tubes sealed at both ends till they are used. Cut one end with file when required.

4.4 Thermometer — of suitable range and accuracy 0·1°C and having graduations at every 0·1°C (*see* IS : 4825-1968*).

4.5 Magnifying Glass — if required.

5. PROCEDURE

5.1 Material Existing as Solids at Ordinary Temperatures

5.1.1 *Preparation of Samples* — Take 2 to 5 g of the dry material and grind in a clean dry porcelain mortar. Take a part of this ground sample and reduce to fine powder in an agate mortar. If the sample consists of only a

*Specification for laboratory and reference thermometers.

very small amount of material, the whole of it may be ground directly in an agate mortar, and if the sample is unduly large or coarse, a preliminary reduction to a suitable size is carried out.

5.1.2 Transfer a sufficient quantity of the dried powder to a clean, dry capillary tube and pack the powder by tapping the tube on a hard surface or by rubbing the outside of the capillary tube with a file so as to make a compact column of about 5 mm high. Fill the heating bath or the Thiele's tube with the heating medium to the required depth, insert the thermometer to the proper immersion depth and heat the bath with constant stirring. When the temperature of the bath is 20°C below the expected melting point, adjust the rate of heating so that the rise in temperature is 2°C per minute. At 10°C below the expected melting point attach the filled capillary tube to the thermometer such that the sample is in close proximity to the thermometer bulb. A rubber band is generally used for this purpose. Continue heating at the rate of 2°C per minute with constant stirring. Record either the melting point or the melting range. Record also the temperature of the emergent stem of the thermometer at the point approximately midway along the merury thread in the emergent stem. If the recorded melting point is less than 7°C above the temperature, at which the sample was inserted, repeat the determination inserting the sample into the bath at a correspondingly lower temperature.

Alternatively electrically heated apparatus may also be used for determination of melting point and melting range. The filled capillary is introduced into a metal block along with the thermometer and the melting point or range is found out. The rate of heating of the metal block may be adjusted by a transformer. The reading is observed through a magnifying glass provided.

5.2 Materials Existing as Liquids at Ordinary Temperatures — Fill a clean, dry 200 × 25 mm boiling tube with the material to a depth of about 50 mm. Insert a suitable thermometer and a glass stirrer into the tube. Immerse the tube with contents in a suitable cooling bath. The cooling bath may consist of ice and water, or of solid carbon dioxide and alcohol, or any other suitable cooling mixture. Stir the contents of the tube well so that bulk of the material has solidified. Take out the tube from cooling bath, dry the outside of it and place it conveniently in a wider boiling tube about 50 mm in diameter to serve as an air-jacket. Immerse the entire assembly in a bath containing a liquid at a temperature about 5 deg above the expected melting point. Stir the material vigorously with the stirrer taking care to break up all lumps and note the temperature at which the last crystal disappears. This temperature is taken as melting point of the material. Record also the temperature of emergent stem, as described in **5.1.2**.

5.3 Calculation — Correct the observed temperature for any error in the calibration of the thermometer and, if required, for the difference in the emergent stem temperature under conditions of calibration and of use, employing the following equation:

$$t_c = 0{\cdot}000\,16n\,(t_s - t_d)$$

where

t_c = correction to be applied to the observed temperature of the melting point,

n = number of degrees celsius on the exposed stem,

t_s = temperature in °C of the emergent stem when standardized, and

t_d = temperature in °C of the emergent stem at the observed melting point.

Report the corrected temperature to the nearest 0·1°C as the melting point or the melting range of the material.

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

Quantity	*Unit*	*Symbol*
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

Quantity	*Unit*	*Symbol*
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

Quantity	*Unit*	*Symbol*	*Conversion*
Force	newton	N	1 N = 0·101 972 kgf
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m²
Frequency	hertz	Hz	1 Hz = 1 c/s (s^{-1})
Electric conductance	siemens	S	1 S = 1 A/V
Pressure, stress	pascal	Pa	1 Pa = 1 N/m²

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AMENDMENT NO. 1 OCTOBER 1988
TO
IS : 5762 - 1970 METHODS FOR DETERMINATION OF MELTING POINT AND MELTING RANGE

(*First cover page, pages* 1 *and* 3, *title*) — Substitute the following for the existing title:

'*Indian Standard*

METHODS FOR DETERMINATION OF MELTING TEMPERATURE/RANGE'

(*Page* 3, *clauses* **1.1** *and* **1.1.1**, *lines* 1 *and* 2) — Substitute 'melting temperature/range' *for* 'melting point and melting range'.

(*Page* 3, *clause* **2.1**, *heading*) — Substitute **'Melting Temperature** *for* **'Melting Point'**.

(CDC 1)

Printed at Swatantra Bharat Press, Delhi, India

AMENDMENT NO. 2 JULY 1990
TO
IS 5762 : 1970 METHODS FOR DETERMINATION OF MELTING TEMPERATURE/RANGE

(*Page* 5, *clause* 5.2) — Insert the following note after 5.2:

'NOTE — For different temperatures, the following cooling media may be used:

a) Ice and water for temperature down to +1°C,

b) Crushed ice and common salt (Nacl) for temperature down to —12°C,

c) Crushed ice and ammonium chloride or calcium chloride ($CaCl_2 . 6H_2O$) for temperature down to—26°C, and

d) 1, 1, 1 trichloroethane and solid carbon dioxide for temperature down to —50°C

(CHD 1)

Printed at Printwell Printers, Delhi, India